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The design and testing of a pulsed sieve plate extraction column

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By

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THE DESIGN AND TESTING OF A PULSED SIEVE PLATE EXTRACTION COLUMN

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(Date)

Professor in Charge

Head of the Department

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ABSTRACT

A pulsed sieve plate extraction column 1 inch in diameter with 24 plates having a free area of 22% and spaced 2 inches apart was constructed and tested on the system water-acetic acid-methyl isobutyl ketone.

Flooding characteristics and performance data were determined at various pulse amplitude-frequency products for the system operated with the ketone phase continuous. Overall plate efficiencies of up to 14% were observed; this is in agreement with data reported by other investigators.

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INTRODUCTION

A pulsed sieve plate column is essentially a convential plate column with smaller holes and without downcomers, but with the addition of a pulse generator. This pulse generator alternately surges the contents of the column in an up and down motion. On the proper stroke of the pulse the dispersed phase is propelled through the continuous phase from one plate to the next. That is, if the light phase is dispersed it goes up from one plate to the next on the upward stroke of the pulse, and if the heavy phase is dispersed it goes down one plate on the downsurge of the pulse.

The main advantage of the pulsed column over the conventional type of packed or sieve tray column is the reduction in contact area made possible by its increased efficiency. This increased efficiency is brought about by the greater interfacial area which the pulsing produces, and by the more thorough intra-phase mixing which reduces considerably the possibility of concentration gradients within each phase. Another property of the pulsed sieve plate column which may be advantageous in certain instances is the fact that no countercurrent flow is possible unless a pulse is applied. Therefore the column may be shut down by stopping the feed streams and the pulser, leaving two

separate phases on each tray. The advantage is that when the column is restarted less time is required to reestablish equilibrium.

The major disadvantage of the pulsed column is the increase in initial cost which is brought about by the necessity for some type of pulse generator. Also, pulsing cannot be used with those systems which tend to form emulsions easily.

The advantage of size reduction which has already been discussed is, of course, apparent, since the initial cost of equipment is usually dependent to a large extent upon its size. However, in the field of nuclear engineering, where the feed streams are usually radioactive, there are two additional reasons for keeping the equipment size down - shielding requirements and the possibility of a criticality accident.

Therefore, it is not surprising that the introduction of pulsing in liquid-liquid extraction processes is primarily the result of work done in the field of atomic energy. For this reason, although much of a general nature has been published on the subject, there is a very limited amount of highly technical unclassified information available on plant scale pulsed columns.

Two plants using full sized pulse columns which have been described in the literature are the U. S. Atomic

Energy Commission's Fernald, Ohio plant operated by the National Lead Company (1), and the AEC's Oak Ridge, Tennessee plant operated by the Union Carbide Nuclear Company (2)

There is a considerable wealth of information available on laboratory scale pulse columns, however. The system used in this study, water-acetic acid-methyl isobutyl ketone, is the one most frequently used in evaluating pulse columns, and results are available for both sieve plate and packed columns.

Results of this study are compared with those of Belaga and Bigelow (3) and Chantry, Von Berg, and Wiegandt (4) in the section on Performance Tests.

DESIGN CONSIDERATIONS

A schematic diagram of the entire system is shown in Figure 2.

The design of the pulse column was based on the completely arbitrary decision to use a column of 1 inch diameter. This was the smallest diameter which had been used by previous investigators (5,6), and therefore represented the optimum safe size for reasonably large throughputs in units of $\frac{cc}{hr.ft^2}$ with fairly small requirements for storage of feed and product.

The contacting section of the column was made 4 ft. high, since this was a standard length and representative of columns reported in the literature. In order that the action of the pulse might be clearly seen during the extraction operation, glass pipe was chosen as the column construction material. End sections were 1 in.-2 in. glass bell reducers 6 inches long, chosen because they were standard sizes easily available and had proved adequate in previous investigations.

The sieve trays were made from 18 gauge stainless steel with 0.067 in. holes as shown in Figure 1. The holes represent 22% of the plate area, a figure which is generally accepted as being the optimum free area. The plates are strung on a 1/8" stainless steel rod and separated from each other by spacers 2 inches long made of 1/4" stainless steel tubing.

With the column details decided upon, the pump and rotameter requirements were determined from the following equation given by Edwards and Byers (6) for incipient flooding due to inadequate pulsing:

(Eq. 1) $$\frac{\pi L}{G} + \frac{\pi}{2} = \frac{\cosh \theta}{\theta}$$

L = heavy (aqueous) phase flow rate $\frac{cc}{min}$

G = light (organic) phase flow rate $\frac{cc}{min}$

$\theta = \frac{G}{\pi Vp}$

Vp = pulsed volume velocity $\frac{cc}{min}$ (amplitude $\frac{cms}{cycle}$ x frequency $\frac{cycles}{min}$ x column area cm^2)

In order to determine the pulsed volume velocity for use in this equation a value of 60 $\frac{cycles}{min}$ was chosen for the frequency because this is the usual frequency used at Oak Ridge (7). The largest pulse amplitude used at Oak Ridge, 0.9 in., was chosen in order to make Vp and, therefore the feed rates, as large as possible. This minimized the possibility of underdesigning the pumps and flow meters. Then:

$$Vp = 60 \frac{cycles}{min} \; 0.9 \frac{in.}{cycle} \; 2.54 \frac{cm}{in.} \; CSA$$

$$Vp = 60 \frac{cycles}{min} \; 0.9 \frac{in.}{cycle} \; 2.54 \frac{cm}{in.} \; \frac{(2.54)^2}{4}$$

$$Vp = 693 \frac{cc}{min}$$

Since both flow rates were unknown, it was necessary to assume some relationship between them. For the purpose of this calculation, it was assumed that both rates were equal, that L = G.

Equation (1) then became:

$$\frac{3}{2}\pi = \frac{\cosh\theta}{\quad}$$

$$\cosh\theta = 4.71\,\theta$$

$$\theta = 0.215$$

Since: $$\theta = \frac{G}{\pi\, Vp}$$

and: $$Vp = 693\ \frac{cc}{min}$$

$$0.215 = \frac{G}{693\,\pi}$$

$$G = L = 417\ \frac{cc}{min}$$

The feed pump chosen was a Milton Roy Duplex Proportioning Pump with a 5/8" diameter plunger, a continuously adjustable stroke length from 0-3 in. on each side, and a maximum capacity of 500 $\frac{cc}{min}$ per side. The rotameters were made by the Brooks Rotameter Company and also had capacities of 500 $\frac{cc}{min}$.

The feed and product tanks were 10 gallon stainless steel tanks which were large enough to permit continuous runs of about 2 hours at 75% of the theoretical flooding throughput. All feed and product lines were 1/4 in. stainless steel tubing.

The pulse generator chosen was a Milton Roy Simplex, Controlled Volume Pump with a 5/8 in. diameter plunger, a continuously adjustable stroke length from 0-3 inches, and a continuously adjustable drive which permitted frequency adjustments of from 0-90 cycles/min. This piece of equipment was essentially a positive displacement pump and was converted to a pulse generator by removal of the check valves and plugging of the inlet port. The pulse it generated was always sinusoidal.

Light and heavy phase feeds were introduced at points just below the bottom plate and just above the top plate, respectively. The pulse was impressed on the heavy phase process solution at a point in the end section about 4 inches below the bottom plate. Effluent streams were removed at the very ends of the calming sections so that the phases might have as long a period as possible for disengaging.

Pulsations in the feed streams caused by the action of the feed pumps rather than the pulser were removed by surge tanks located between the pumps and the rotameters.

The location of the principle interface which determined which of the two phases was continuous (organic if at the bottom, aqueous if at the top) was set by the height of a vented teflon jackleg on the aqueous effluent line. This effluent line was also fitted with a check valve to prevent liquid from draining out of the jackleg on the down surge of the pulser.

FIGURE 1 DIAGRAM OF SIEVE TRAY

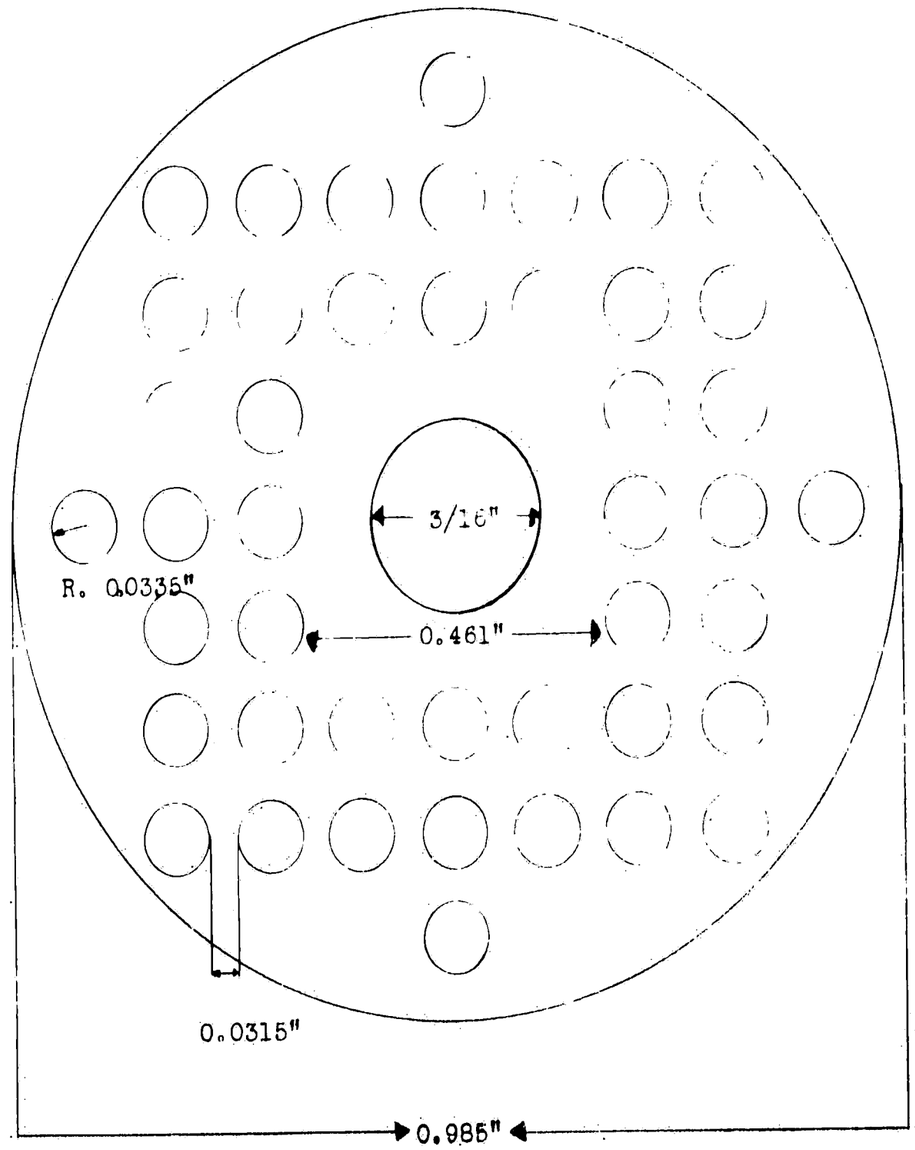
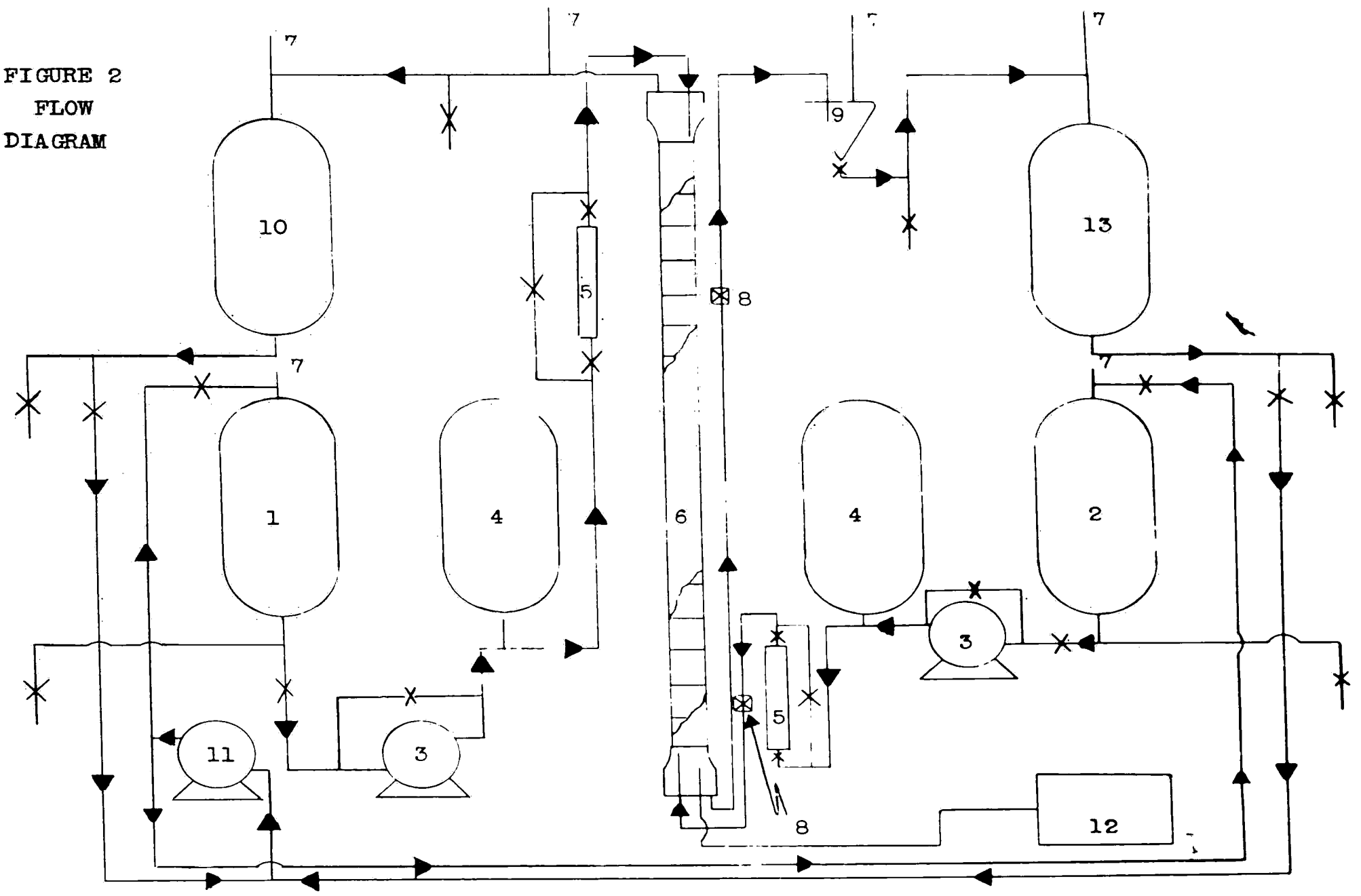


FIGURE 2
FLOW
DIAGRAM



KEY TO FIGURE 2

1 - Aqueous feed tank

2 - Organic feed tank

3 - Duplex feed pump

4 - Surge tanks

5 - Rotameters

6 - Pulse column

7 - Vents

8 - Check valves

9 - Vented tank at top of jackleg

10 - Organic product tank

11 - Materials handling pump for any transfer necessities that may arise

12 - Pulse generator

OPERATING PROCEDURE

In order to begin operation when the column was emptying, the feed pumps were turned on, the aqueous feed stroke length adjustment dial set at 100, the organic feed dial set at 0, and the black-handled valves beneath the rotameters opened wide. When flow was indicated on the aqueous feed rotameter, the aqueous feed dial was set for the desired flow rate. Most of the aqueous feed which entered the column remained in the upper calming section, but a small amount trickled down the walls of the column and through the holes in the plates to form a puddle in the lower end section. When the liquid level in the lower end section covered the tube through which the pulse was impressed, the pulse generator was turned on, the desired amplitude set on the dial and the proper frequency set on the speed control. (Note: The speed control must not be adjusted unless the motor is running.) The action of the pulse in the column was enough to permit the aqueous feed to displace the air and the column began to fill rapidly.

As soon as the pulse was adjusted, the organic feed dial was set at 100. When flow was noted on the organic feed rotameter, the organic feed dial was set for the desired flow, and the column allowed to fill.

The principle interface was adjusted so that it was in the lower end section by means of the flexible jackleg (it could have been maintained in the upper end section if the jackleg had been longer). The column was now operating properly and was permitted to reach equilibrium - a process which generally took about 30 minutes. During this period, it was often necessary to adjust the height of the interface, because the density of the material in the column changed as the system approached equilibrium.

While the system was equilibrating, samples were taken from each feed tank and titrated with 0.5N N_aOH. A 5 ml aliquot was used for the aqueous feed and a 50 ml aliquot for the organic feed. Feed densities were determined on a Westphal balance.

At the end of 30 minutes, a sample of each product stream was taken and titrated with 0.5N N_aOH. In order that a material balance might be made, a 50 cc volume of each product stream was timed with a stop watch. After 10 minutes, this process was repeated on the organic effluent stream. If the two values agreed, aqueous samples were taken and this condition accepted as equilibrium. If the samples did not agree, the system was permitted to run for 10 minutes and the process repeated until agreement of 2 consecutive samples was reached. 5 ml aliquots of both streams were used for titrations and densities of all samples were determined on the Westphal balance.

If desired, changes in flow rates and pulse characteristics were made on the system without shutting it down. It was only necessary to repeat the process for reaching and determining equilibrium.

When the column was shut down, the feed pumps were turned off, the black-handled valve on the panel board closed, and the pulse generator stopped. If the columns were to be restarted with the same feed, it was not drained, but when draining was necessary, it was done through the flexible jackleg by removing the stopper from the small vented tank at the top of the jackleg and lowering that part of the jackleg connected to the stopper so that the column drained by gravity into some container.

PERFORMANCE TESTS

Two different types of tests, flooding and extraction, were made on the column.

In a pulsed column, flooding can occur for one of three reasons:

(1) Excessive throughput

(2) Excessive pulsation

(3) Inadequate pulsation

Excessive throughput causes flooding in the column regardless of the pulse amplitude or frequency, and is characterized by the visible entrainment of large droplets of one phase in the effluent line intended for the other. Some slight entrainment of a wrong phase in an effluent line was not considered to be flooding; at the flooding point the presence of the wrong phase in an effluent line was unmistakable. At any throughput lower than that at which total flooding occurs, there is arange of pulse amplitude-frequency products within which the column operates satisfactorily without flooding. Neither pulse amplitude nor frequency alone is controlling, but it is the product of the two expressed as pulse volume velocity which is important (6).

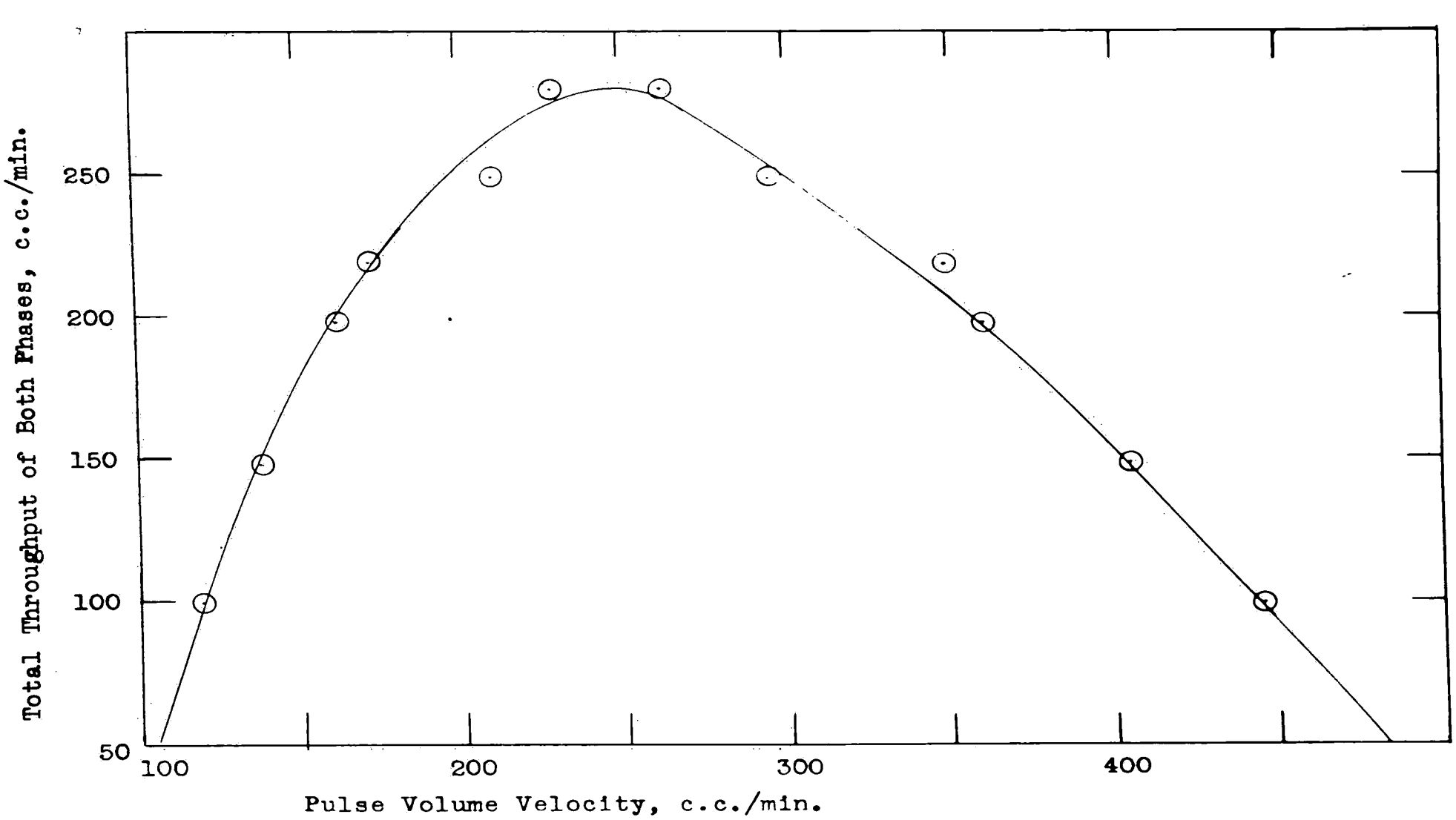
When the column floods because the pulse is excessive, the result is exactly the same as that which occurs during flooding due to excessive throughput.

On the other hand, flooding due to inadequate pulsing is characterized by a slow recession of the principle interface downward in the column, and a simultaneous build-up of the heavy phase in the region above the top plate.

The flooding characteristics of the column used in this study are shown in Figure 3.

The results of the extraction tests made on the column are shown in Figure 4. These agree quite well with the data of Chantry, Von Berg, and Wiegandt (4) for their sieve plate column, and the shape of the curves agree in general with the more extensive results obtained from a packed column by these same investigators. That is, they found that the H.E.T.S. in their packed column reached an almost constant value when plotted against throughput at a point just below flooding. In this study, the overall stage efficiency approached a constant value at the higher throughputs, a condition equivalent in a sieve plate column to that observed by Chantry, Von Berg and Wiegandt (4) in a packed column.

FIGURE 3 FLOODING CHARACTERISTICS



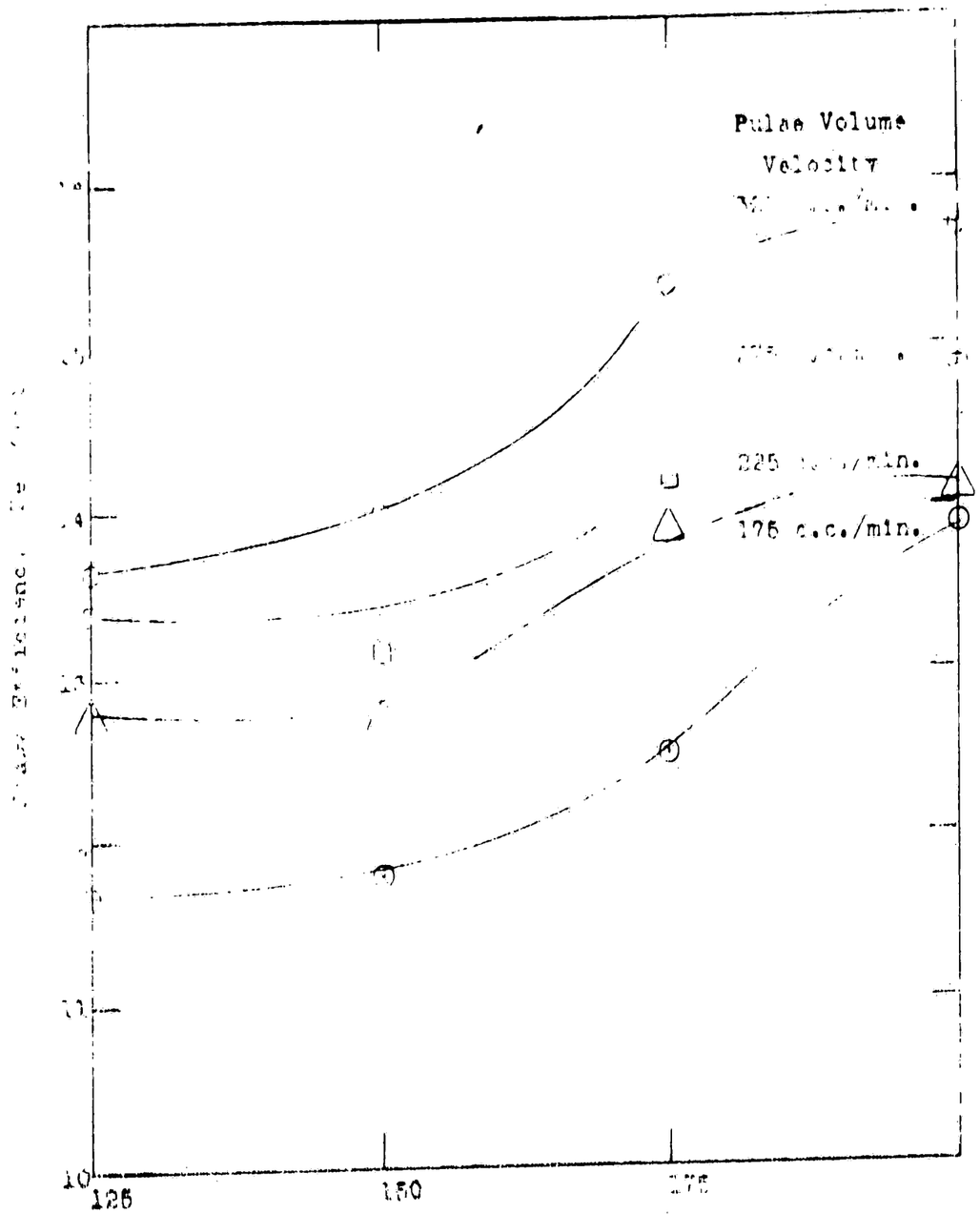


FIGURE 4 EFFECT OF THROUGHPUT AND PULSE ON COLUMN EFFICIENCY

TABLE I

Operating Data

Acid Concentrations, Wt. % HAC				Feed Rates $\frac{cc}{min}$		Pulse		Material Balance		
Ketone Feed	Aqueous Feed	Ketone Effluent	Aqueous Effluent	Ketone	Aqueous	cycles/min.	amp. inches	wt.out / wt. in	No.of Theo. Stages	Stage efficiency,%
0	19.50	12.46	7.00	62.5	62.5	21.5	0.665	1.068	2.809	11.70
0	19.50	12.82	6.96	62.5	62.5	27.8	0.665	1.062	3.077	12.80
0	19.50	13.29	6.85	62.5	62.5	33.7	0.665	1.068	3.218	13.40
0	19.50	13.39	6.72	62.5	62.5	39.7	0.665	1.070	3.271	13.62
1.04	16.51	11.06	7.06	87.5	87.5	21.5	0.665	1.002	3.000	12.50
1.04	16.51	11.35	6.79	87.5	87.5	27.8	0.665	0.998	3.333	13.89
1.04	16.51	11.46	6.67	87.5	87.5	33.7	0.665	0.994	3.401	14.19
1.04	16.51	11.37	6.76	87.5	87.5	39.7	0.665	0.994	3.686	15.39
0.92	19.19	11.75	6.97	75.0	75.0	21.5	0.665	1.111	2.834	11.79
0.92	19.19	12.06	6.88	75.0	75.0	27.8	0.665	1.099	3.048	12.72
0.92	19.19	12.19	6.79	75.0	75.0	33.7	0.665	1.095	3.154	13.12
0.92	19.19	12.29	6.64	75.0	75.0	39.7	0.665	1.095	3.306	14.00
0.89	23.40	15.32	7.49	100.0	100.0	21.5	0.665	0.920	3.340	13.92
0.88	23.38	15.34	7.41	100.0	100.0	27.8	0.665	0.919	3.392	14.12
0.88	23.38	15.70	7.32	100.0	100.0	33.7	0.665	0.914	3.565	14.86
0.88	23.38	15.88	7.21	100.0	100.0	39.7	0.665	0.919	3.770	15.71

SUGGESTED EXPERIMENTAL PROGRAM

It is recommended that extraction studies be made using the water-acetic acid-methyl isobutyl ketone system with the aqueous phase continuous. Greater extraction efficiency should probably result (4).

At some future time, studies may be conducted using different plate spacings or geometries to determine the effect on stage efficiency.

Since all tests conducted, thus far, have used equal volumetric flow rates of the phases, it may be worth while to vary one of the flow rates to determine the effect on efficiency.

CALIBRATIONS

Calibration curves for the feed rotameters and the feed pump stroke length adjustment dials are given in Figures 5, 6, 7, and 8.

The calibration curve for the speed control on the pulse generator is given in Figure 9.

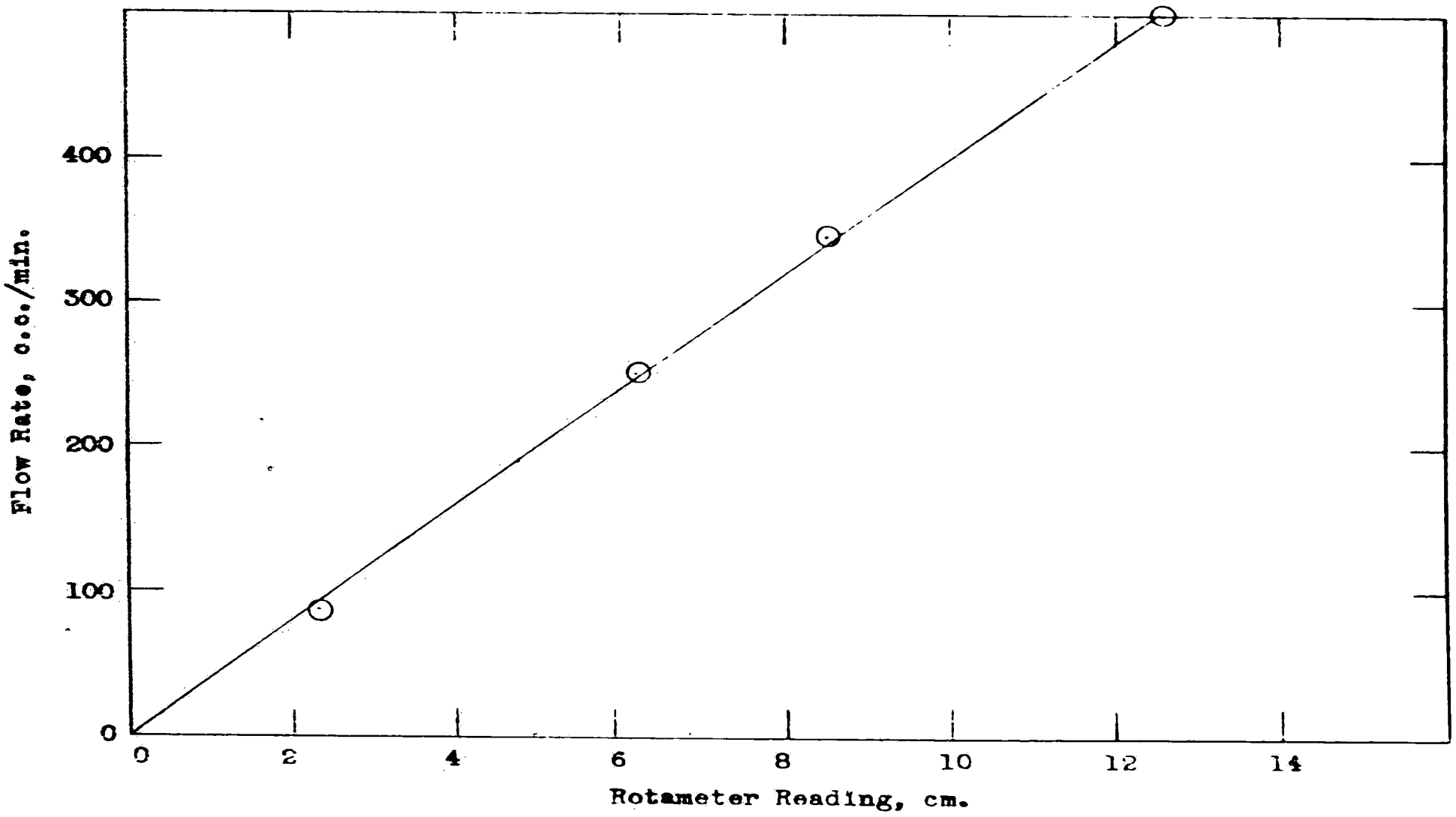


FIGURE 5 ORGANIC PHASE ROTAMETER CALIBRATION

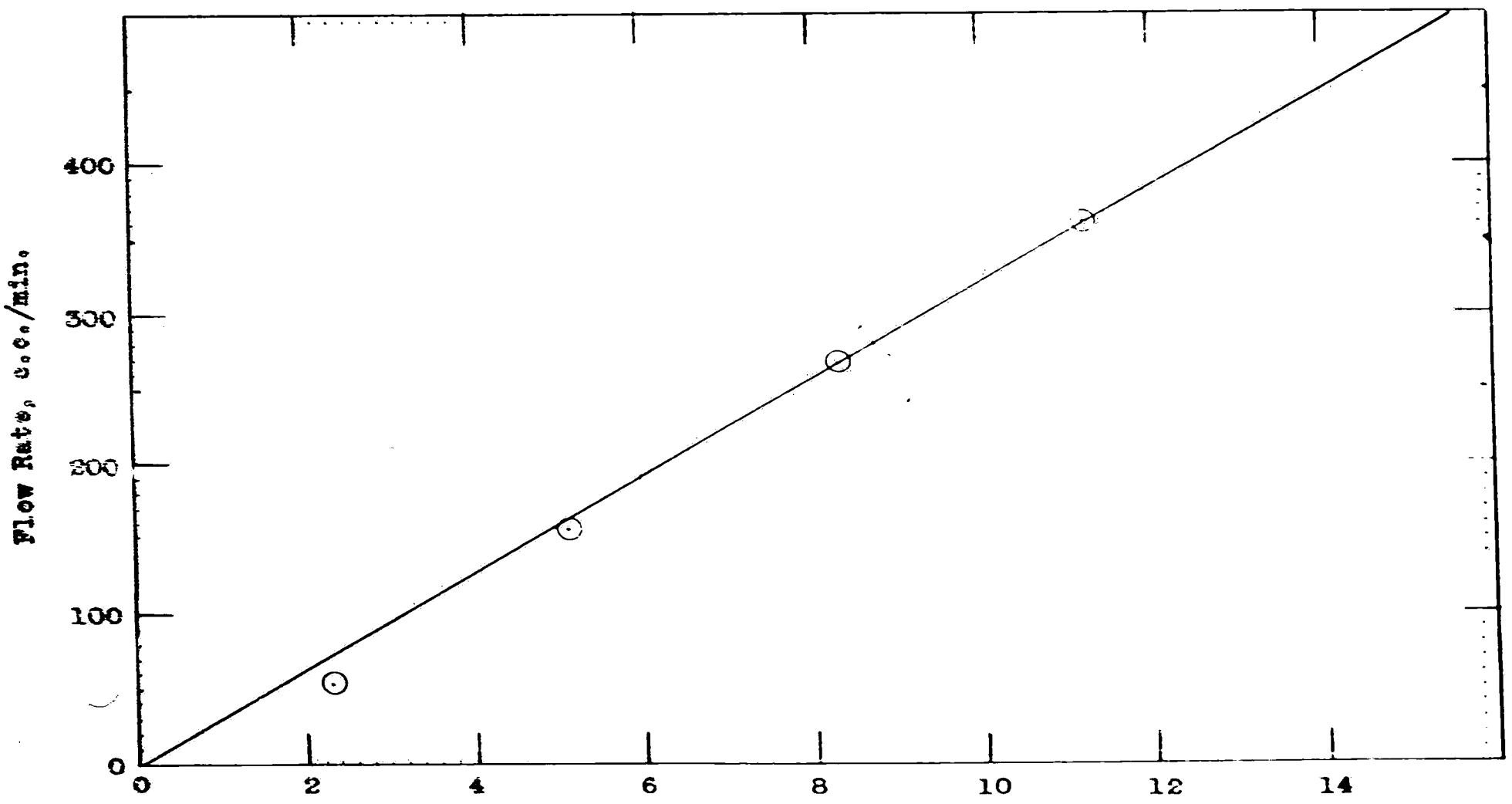


FIGURE 6 AQUEOUS PHASE ROTAMETER CALIBRATION

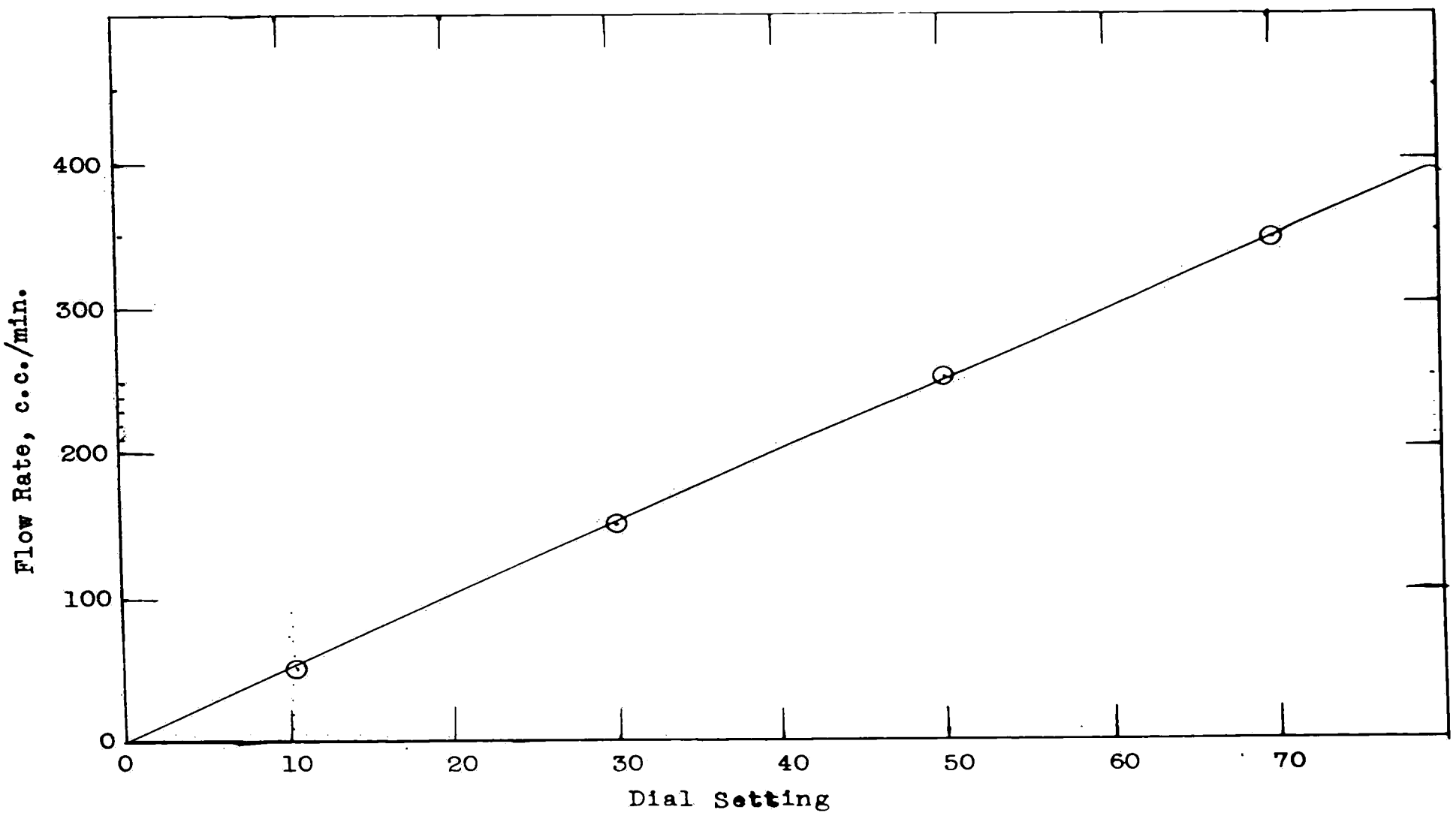


FIGURE 7 CALIBRATION OF ORGANIC PUMP DIAL

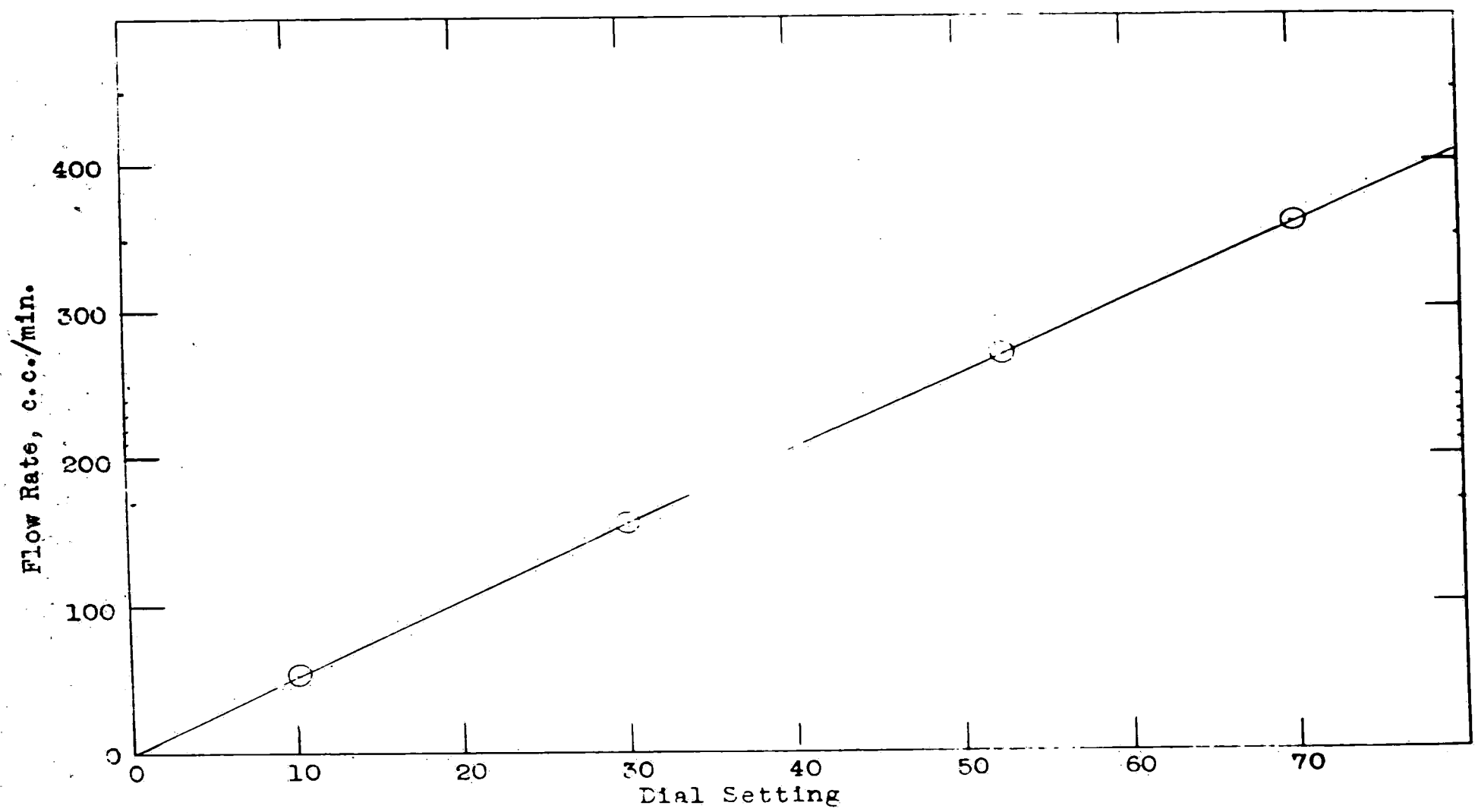


FIGURE 8 CALIBRATION OF AQUEOUS PUMP DIAL

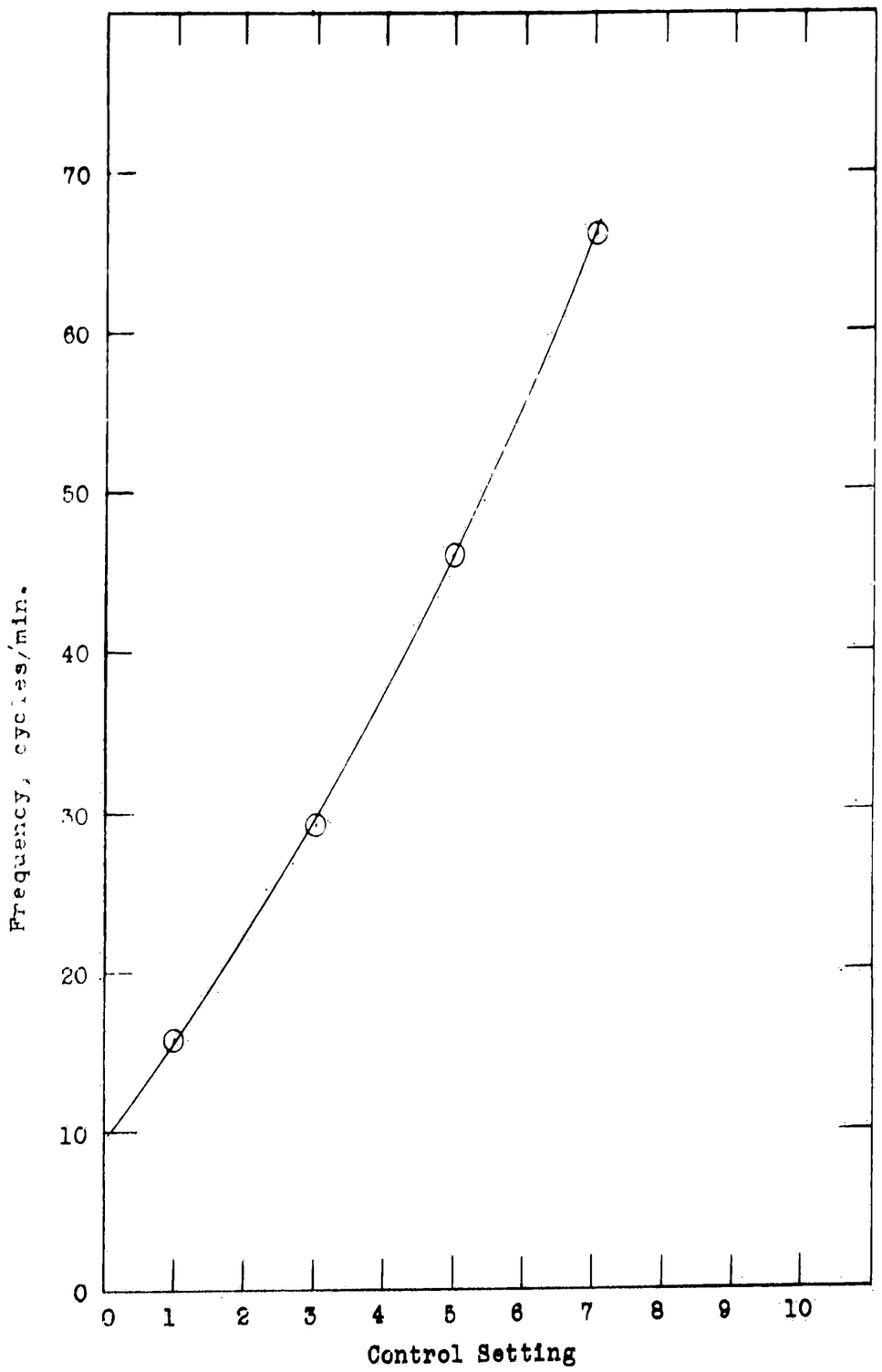


FIGURE 9 PULSE FREQUENCY CONTROL CALIBRATION CURVE

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CALCULATION

The percentage of acetic acid in any stream was determined from the following equation:

$$\% \text{ HAC} = \frac{V N}{V'} \quad 60.02 \quad \frac{1}{\rho} \quad \frac{1}{10}$$

V = Volume of N_aOH used in titration (cc's)

N = Normality of N_aOH used in titration

V' = Volume of acid sample titrated (cc's)

ρ = Density of acid sample $\frac{\text{gms}}{\text{cc}}$

Example:

The aqueous feed used at the flow rate of 87.5 $\frac{\text{cc}}{\text{min}}$ per phase gave the following data:

Sample (mls)	Vol. NaOH (mls)	Sp gr.	Normality of N_aOH
5	27.9	1.0182	0.5006

Therefore:

$$\% \text{ HAC} = \frac{(27.9)(0.5006)}{(5)} \quad 60.02 \quad \frac{1}{1.0182} \quad \frac{1}{10}$$

$$\% \text{ HAC} = 16.51$$

The material balance for each run was determined by dividing the measured total weight of material leaving

by the total weight of material entering as indicated by the feed pump stroke length adjustment dials.

The total weights entering and leaving were determined as follows:

$$\text{Weight leaving} = \frac{V_A}{\theta_A} \; 60 \; \rho'_A + \frac{V_o}{\theta_o} \; 60 \; \rho'_o \quad \frac{\text{gms}}{\text{min}}$$

V_A = Volume of aqueous sample taken in cc's

θ_A = Time in sec. required to take aqueous sample of volume V_A

ρ'_A = Density of the aqueous sample taken in $\frac{\text{gms}}{\text{cc}}$

V_o = Volume of organic sample taken in cc

θ_o = Time in sec. required to take organic sample of volume V_o

ρ'_o = Density of organic sample taken in $\frac{\text{gms}}{\text{cc}}$

$$\text{Weight entering} = (L)\,(\rho_o) + (N)\,(\rho_A) \quad \frac{\text{gms}}{\text{min}}$$

L = Organic feed rate in $\frac{\text{cc}}{\text{min}}$

ρ_o = Density of organic feed in $\frac{\text{gms}}{\text{cc}}$

N = Aqueous feed rate in $\frac{\text{cc}}{\text{min}}$

ρ_A = Density of aqueous feed in $\frac{\text{gms}}{\text{cc}}$

Example:

Run No. 5 had the following data:

Ketone feed rate	Aqueous feed rate	Density of Ketone feed	Density of Aqueous feed	Sec/50 cc Aqueous effluent
87.5	87.5	0.8052	1.0182	54.8

Sec/50 cc Ketone effluent	Density of Ketone effluent	Density of Aqueous effluent
23.9	0.8372	1.0057

$$\text{Weight leaving} = \frac{50}{54.8}\ 60\ 1.0057 + \frac{50}{23.9}\ 60\ 0.8372$$

$$\text{Weight leaving} = 160.1\ \frac{\text{gms}}{\text{min}}$$

$$\text{Weight entering} = (87.5)\ (0.8052) + (87.5)\ (1.0182)$$

$$\text{Weight entering} = 159.5\ \frac{\text{gms}}{\text{min}}$$

$$\text{Material balance} = \frac{\text{Weight leaving}}{\text{Weight entering}} = \frac{160.1}{159.5} = 1.002$$

The determination of the number of theoretical stages was made graphically, and an example of this is shown in Figure 10. The overall stage efficiency for each run was determined by dividing the number of theoretical stages by the number of actual stages, 24, and multiplying by 100.

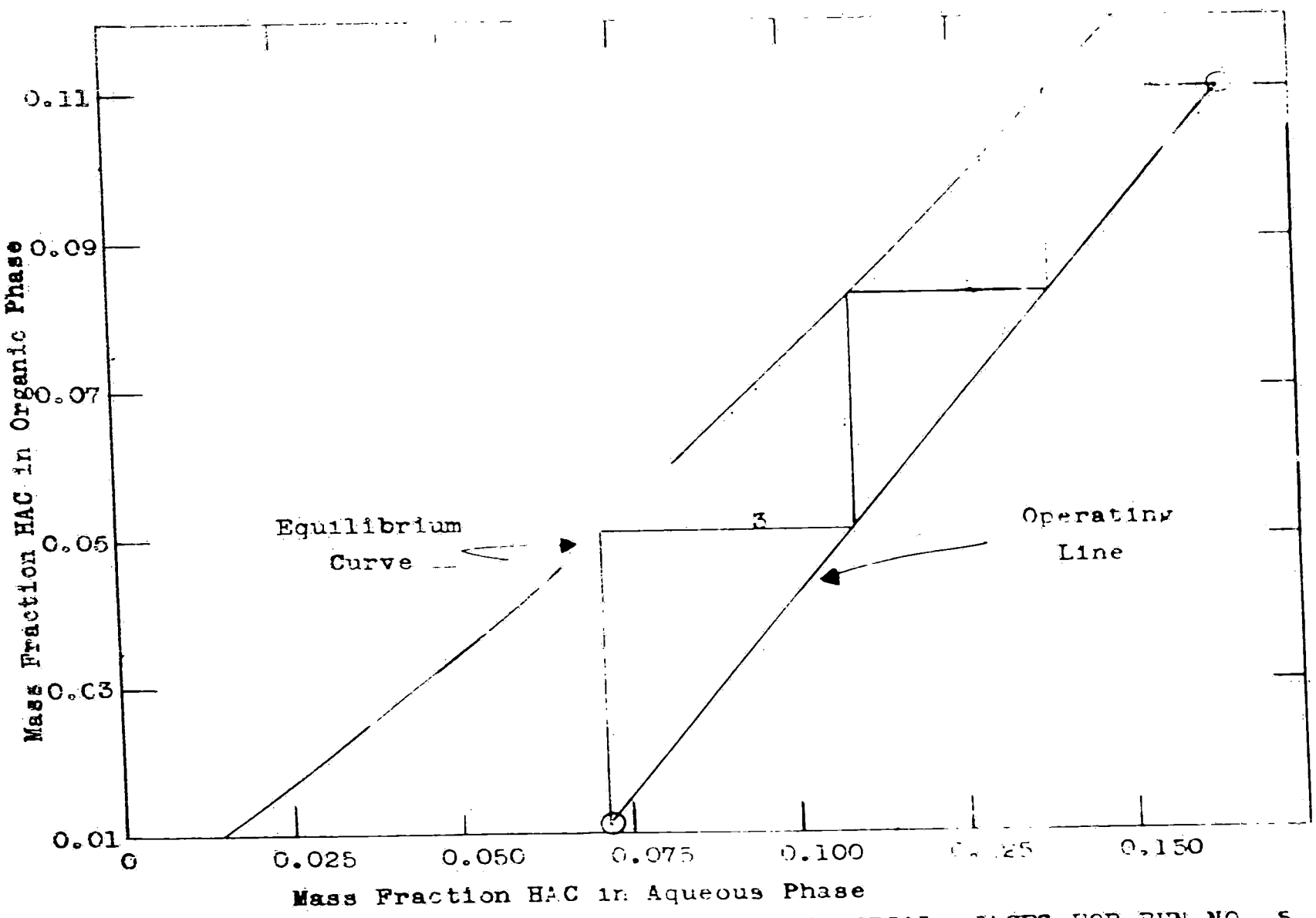

FIGURE 10 DETERMINATION OF NUMBER OF THEORETICAL STAGES FOR RUN NO. 5

VITA

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He attended the Providence public school and the University of Rhode Island, receiving his B.S. in 1956.